PATENT

#9

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re:	Kendall, et al.	**Docket No.:**	IR-2795(EC)
Serial No.:	09/888,793	**Filing Date:**	6/25/2001
Examiner:	Robert D. Harlan	**Art Unit:**	1713
For:	"Metathesis Polymerization Adhesives and Coatings"		

****

Nov. 22, 2002

Assistant Commissioner for Patents
Washington, DC 20231

DECLARATION PER 37 C.F.R. 1.131

Sir:

This is in response to the Office Action dated May 22, 2002. The undersigned Applicants declare the following:

As a below named inventor, we hereby declare that:

Our residence, post office address and citizenship are as stated below next to our names; and

we believe we are the original, first and joint inventors of the subject matter which is claimed and for which a patent is sought on the invention entitled, "Metathesis Polymerization Adhesives and Coatings" the specification of which was filed 6-25-01 in the United States Patent and Trademark Office.

Submitted as attached is objective evidence of invention by Applicants prior to the reference effective date, December 5, 2000 of Japanese Kokai Patent Application 336320 as attached photocopy pages of Notebooks and is evidence of actual reduction to practice, summarized as follows:

Pages 28 and 29 from Notebook No. 8148, evidencing that polymerization between rubber and metal with bonding was achieved using Grubb's catalyst and ENB monomer.

Pages 58 and 59 from Notebook No. 8060 showing that Grubbs catalyst coatings on EPDM substrate followed by coating of p-ENB resulted in a polymerized coating; and catalyst coatings on glass slides followed by spraying of ENB monomer resulted in a peelable solid coating film.

Page 57 from Notebook No. 8148 showing that monomer mixtures of ENB and TCHP polymerized with Grubb's catalyst.

Pages 50 and 51 of Notebook No. 8297 demonstrated contact metathesis adhesion between different polyolefin (LDPE) substrate bonded to itself with 5,5-bis(chloromethyl)-2-norbornene using Grubb's catalyst.

Pages 41 and 42 of Notebook No. 8327 demonstrated the bonding of polypropylene to itself using 5,6-bis(chloromethyl)-2-norbornene delivered from a 2-part cartridge.

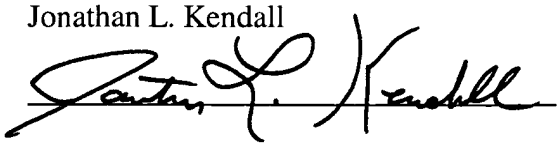
Pages 66 and 67 of Notebook No. 8327 demonstrated bonding of polypropylene using ENB and Grubb's catalyst.

Pages 64 to 65 of Notebook No. 8363 demonstrated formulation as a two part adhesive of ENB, NBD, silica, and an elastomer (Europrene) provided a measured amount of bond strength in lap shear samples.

Pages 11, 12, 15, 22 and 23 of Notebook No. 8374 demonstrated formulation as a two part adhesive of norbornadiene, ENB, Blendex, Cab-o-sil and Zeeospheres provided a measured bond strength in lap shear polypropylene samples.

Pages 51 to 56, 59-60 and 89-90 of Notebook No. 8374 demonstrated 2-part adhesive formulations applied to bond polypropylene substrates.

We further declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of first inventor:	Jonathan L. Kendall
Inventor's Signature:	
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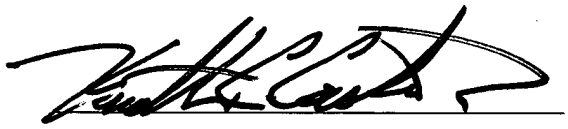
Inventor's Signature:	
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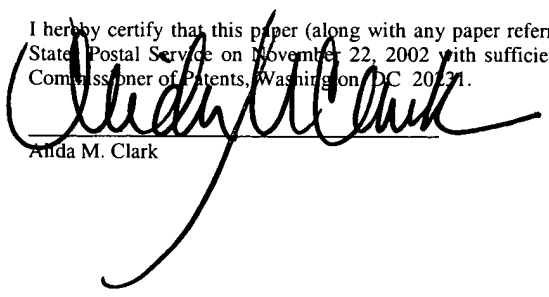
CERTIFICATE OF MAILING (37 CFR 1.8(A))

I hereby certify that this paper (along with any paper referred to as being attached or enclosed) is being deposited with the United States Postal Service on November 22, 2002 with sufficient postage as first class mail in an envelope addressed to the Assistant Commissioner of Patents, Washington, DC 20231.



Alida M. Clark

11/22/02

Date

Project No.

Book No. 8148 TITLE

From Page No. [redacted]

Rubber to Metal Bonding Using Monomer / Catalyst Mixture

Materials: ENB (CK8148-15-1); Grubbs's Catalyst (Strem); EPDM (J. Taylor); Grit Blasted Metal coupons (M. Righettini)

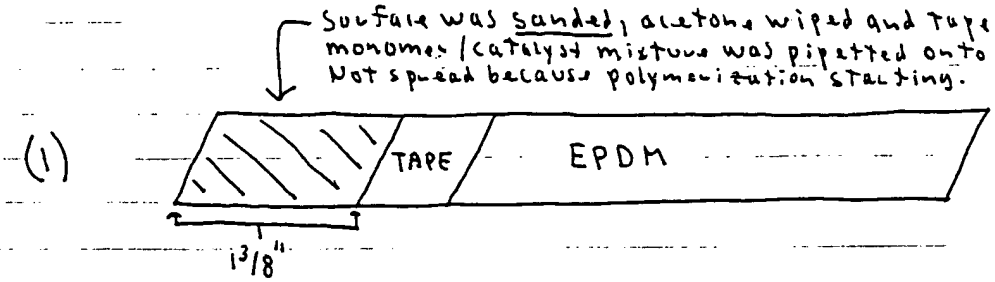
Procedure:

1) 0.0009g of catalyst (0.000001 moles) was weighed into a glass vial.

2) 2.8ml (2.5004g or 0.0208 moles) of ENB was quickly syringed into the glass vial and immediately stirred for a few seconds.

3) The monomer/catalyst mixture was then dropped onto the EPDM strip using a glass pipet. The metal coupon was finally placed over the treated EPDM surface and held in place until bonding occurred. A brown jar was placed over the bonded area. Monomer to catalyst molar ratio was 20,800:1.

4) Bonding procedure was as follows (pictured from above description):



Immediately, the acetone washed metal coupon was placed on the EPDM strip in treated area. Material came out the sides and polymerized during bonding. The coupon was held in place until hard to move.

To Page No. 29

Witnessed & Understood by me,	Date	Invented by	Date
[redacted]	[redacted]	Recorded by Chris Kerk	[redacted]

From Page No. 28

5) 5 hrs after bonding, metal easily pulled apart from rubber. Nothing on metal. Rubber had a thin, elastic film stuck to it.

PART II

Steps 1-4 (pages 28) were repeated. However, this time the monomer/catalyst mixture was pipetted onto the metal surface and the EPDM quickly placed onto the metal surface. The reverse of the first experiment. 17 hrs after bonding, metal again easily pulled apart from the rubber. Like above, nothing on metal while rubber had a thin, elastic film stuck to it.

C.K. [redacted]

To Page No. ____

Witnessed & Understood by me,	Date	Invented by	Date
S E [redacted]	[redacted]	R c rded by Chris Kerk	[redacted]

From Pag N .58

Purpose: To prepare samples for surface analysis on CMP technology. Ed Tokas will be taking different samples to NJ w/MAS to conduct ESCA analysis.

Instructions: from Ed Tokas

Printed By: Ed Tokas Page: 1 [redacted] PM

From: Ed Tokas ([redacted])
To: Chris Keck, Russell Walls
CC: Ken Caster, Steve Howe, Marlene Righettini, Lynn Yanyo
REGARDING CMP Samples for Analysis
Chris and Russ,

Giving you a reminder that next week I will be going to conduct two separate surface analyses on CMP samples:

1st Tuesday Marlene and I will go to NCSU to run a cross sectional analysis on R/R bonded samples (one freshly prepared and one aged sample).

2nd On Wednesday I will be going to NJ w/ MAS to conduct ESCA analysis and will need the following samples:

Russell Walls [redacted]

Labeled as Notebook Page	Sample
✓ -1	EPDM (for all preps in this series use the 1/16 in. pads located in my office)
✓ - 2	EPDM w/ catalyst
-3	EPDM w/ a thin layer of poly-ENB (thinnest of three samples)
-4	EPDM w/ intermediate thickness of poly-ENB
-5	EPDM w/ thick layer of poly-ENB
-6 and -7	A free film of poly-ENB (prepared on glass and carefully removed)
-8	A bulk polymerized poly-ENB (prepare in a tube)
✓ -9	Metal coupon (the grit blasted Fe coupons we have been using)
✓ -10	Metal w/ catalyst (the grit blasted Fe coupons we have been using)

See me Monday AM to discuss.

Thanks Ed

Procedure/log

Time	Comments
8:30 am ([redacted])	Discussions with Ed Tokas to make clear what is to be done. Since the samples are to be sprayed, I was instructed to prepare most of the samples. Chris Keck was instructed to make the bulk polymerized poly-ENB in a small test tube.
1:00 pm	• Ed Tokas wanted to be present for first spraying. 0.75g of Lupersol (2) catalyst was dissolved in 10ml CH_2Cl_2 and swirled till dissolved.
1:10 pm wiped with acetone to clean first.	• Coated 1/16" 6"×6" EPDM with catalyst on two passes, dry, and two more passes for a total of 4 passes. Ed said it looked fine. Asked that we wait 30 minutes before applying monomer.

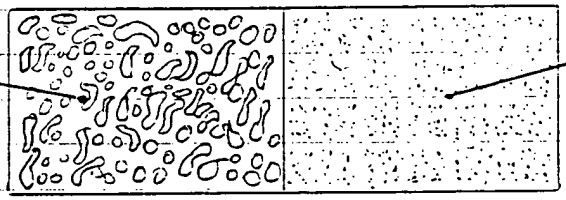
To Pag No. 59

Witness d & Understood by m ,	Date	Invented by	Dat
James B Keck	[redacted]	Recorded by [redacted]	[redacted]

From Page No. 58

[redacted]

Time	Comments
1:40 pm	· Applied thin coat of poly-ENB by making two passes over the rubber surface. Allowed to dry in hood. Smell of monomer still present. 1 sample each just catalyst stored.
1:45 pm	· Acetone wiped EPDM labelled as RW8060-58-1 with silver pen on back and placed in ziplock bag. Bag labelled and brown paper placed in front of surface to be tested so that it would not come in contact with plastic of bag.
	· EPDM with just catalyst labelled as RW8060-58-2 and stored in ziplock bag just like RW8060-58-1.
2:00 pm	· Forgot to note that at 1:40 pm I also coated two small metal coupons (not grit blasted) with 2 mils catalyst. Stored and labelled as RW8060-58-10 and placed in ziplock bag. The unsprayed coupons just wiped with acetone stored and labelled as RW8060-58-9
	· Also at 1:40 pm I coated one micro glass slide with catalyst.
2:20 pm	· Sprayed glass coated slide with monomer (ENB). Solvation occurred to disrupt smoothness of catalyst evenly applied



· Subsequent spraying of monomer done to build thickness. Razor blade at edge indicates peelable coating. Allowed to dry fully before storing and labelling as RW8060-58-6 and -7 in ziplock bag.

3:00 pm · Made another catalyst solution 0.75 gm in 10 ml $MeCl_2$

· We have been learning the glashing on each sample so far. This means that we have coated it as well.

· After wiping surface of RW8060-58-4 sample with acetone I attempted to weigh it to help determine coating weight, but couldn't get accurate reading. The 6"×6" with glashing is to large to weigh with draft shield in place and air currents in lab-5 are too great to get accurate weights. Veeco tester may be used to determine thicknesses.

To Page No. 60

Witnessed & Understood by me,	Date	Invented by	Date
James B Regan	[redacted]	R corded by Russell Wells	[redacted]

From Page No. [redacted]

Catalyst Activity Study Using Tricyclohexylphosphine

Materials: Tricyclohexylphosphine (TCHP, 97% from Aesar), ENB (CK8148-15-1), Grubbs's Catalyst (Boulder)

Procedure:

1) Polymerization with ENB and the Grubbs's catalyst is very fast. TCHP was added to the catalyst in order to slow down the polymerization. Different amounts of TCHP was added to the catalyst and then the ENB syringed into the mixture. Time of polymerization was then recorded. Table of results shown below:

M/I Ratio	TCHP/I Ratio	ENB (ml)	I (gms)	TCHP (gms)	Polymerization Time
5,000:1	2:1	3.0	0.0037	0.0025	1 min.
5,000:1	5:1	3.0	0.0037	0.0063	2 min.
5,000:1	10:1	3.0	0.0037	0.0125	4 min.
5,000:1	Control	3.0	0.0037	—	13 sec.
10,000:1	2:1	3.0	0.0018	0.0013	1 min. 4 sec.
10,000:1	5:1	3.0	0.0018	0.0031	2 min. 15 sec.
10,000:1	10:1	3.0	0.0018	0.0063	4 min. 50 sec.
10,000:1	Control	3.0	0.0018	—	32 sec.

M = Monomer (ENB)
I = Initiator (Grubbs's Catalyst)
TCHP = Tricyclohexylphosphine

To Page No. ____

Witnessed & Understood by m, [redacted] Date [redacted]

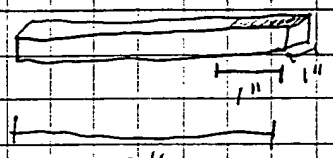
Invented by [redacted] Date [redacted]

Recorded by Chris Keck

From Page No. 50

[redacted]

CMP of polyolefins with
5,6-bis(chloromethyl)-2-norbornene



Polypropylene and LDPE strips were covered, as shown in the picture on the left, with ~0.1mL each of 5,6-bis(chloromethyl)-2-norbornene. The area covered was 1in² at the end of a 6" x 1" strip. Two strips of each substrate type[redacted] were treated. A solution of 10mg Grubbs' catalyst in ~0.1mL 5,6-bis(chloromethyl)-2-norbornene was prepared and quickly applied to one of each substrate types such that the solution was divided evenly between the two substrates. The catalyst soln was spread evenly over the 1in² end of the substrate. The corresponding substrate without catalyst was inverted and placed in contact with the [redacted] catalyst solution. A 200g weight was placed on the top of the substrates & the reaction was allowed to cure for 24h.

To Page No. 51

Witness d & Understood by me,
Amy C. Burke

Date [redacted]

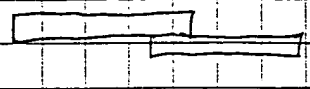
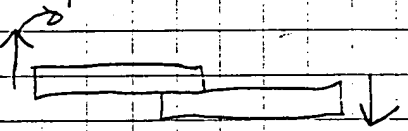
Invented by Justin Kimball

Recorded by Justin Kimball

Date [redacted]

From Page No. 50

[redacted]

After 24h, the weight was removed from the samples & the bond strength was tested by hand. When tested by shear: ←  → The bond could not be broken by hand. When tested under peel conditions  After some slight initial effort, the samples peeled off with ease. That is, once a crack was formed it propagated at the adhesive-substrate interface with ease. The adhesive was a hard plastic-like material.

Peter Kovacs

To Page No. ____

Witnessed & Understood by me, Amy C. Burke

Date [redacted]

Invented by Peter Kovacs

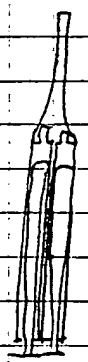
Recorded by Peter Kovacs

Date [redacted]

From Page No.___

Bonding of Polypropylene Using 5,6-bis(Chloromethyl)-2-norbornene, $WCl_4(O\text{-}C_6H_5)_2$, + $SnBu_4$ [redacted] delivered as a 2-part adhesive from a cartrige.

A solution of 136mg (167μmol) $WCl_4(O\text{-}C_6H_5)_2$ [redacted] in 1g 5,6-bis(chloromethyl)-2-norbornene + 40mg poly ENB (synthesized in 8290-p62 sample # 2) was prepared. A solution of 0.05mL $SnBu_4$ in 1g 5,6-bis(chloromethyl)-2-norbornene + 40mg poly ENB (8290, p62, sample #2) was prepared. The solutions were placed in A + B sides respectively of an in-house manufactured 1mL cartrige fashioned from 2 1mL ~~syringes~~ [redacted] syrringes (plastic disposable) + a mixing chamber from the tip of a polypropylene pipette.



The mixing chamber was attached to the syrringes with hot melt polyethylene adhesive.

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Witnessed & Understood by me,	Date	Invent d by	Date
Orian Canelas	[redacted]	Recorded by	[redacted]

From Page No.

The cartridge was used to deliver the adhesive to 6 sanded (w/ 100 grit paper) polypropylene coupons over a 1in² area. The polypropylene coupons were mated with 6 additional sanded polypropylene coupons. The joints were held in place with a 170g weight.

After 24h, 3 samples were tested. Two pulled apart by hand + a third was pulled apart on the speedy tester with a stress at break of 57psi.

After 72h, the 3 remaining samples were tested. Two pulled apart by hand + the third was pulled apart on the speedy tester with a stress at break of 217psi. The samples that pulled apart by hand were not fully cured.

Jonathan Kimball

To Page No.

Witnessed & Understood by me.	Dat	Invented by Jonathan Kimball	Dat
Florian Canelas		Recorded by Jonathan Kimball	

Project No. _____
Book No 8327 TITLE _____

From Page No. ___

Bonding of Polypropylene Samples using EMB + low levels of Grubbs' catalyst.

[redacted]

A solution of 0.5mg (0.6 μmol) Grubbs catalyst in 1g (8.3 mmol) 5-ethylidene-2-norbornene was prepared + quickly applied to one face of a 1" x 4" x 1/8" polypropylene substrate (sanded with 100 grit paper + washed with acetone) over a 1 in² area. The coupons were mated with sanded polypropylene coupons such that a 1 in² ~~except 1/8~~ [redacted] overlap resulted. The solution was viscous within about 30 sec + the 5th coupon pair was joined after some viscosity had built up. The bonds were held in place with a 170g weight. Monomer : Catalyst ratio = 14000 : 1
Samples 8327-66-1, 2, 3, 4, 5.

The procedure ~~above~~ [redacted] above was repeated with 5 additional coupon pairs with the exception that

T Page No. ___

Witnessed & Understood by me,	Date	Invented by	Date
Dorian Canelas	[redacted]	R corded by	[redacted]

From Page No.

0.25 mg (0.3 μmol) Grubbs catalyst was dissolved in 1 g (8.3 mmol) 5-ethylidene-2-norbornene resulting in a monomer:catalyst ratio of 28000:1. As before, the last capon (8327-66-10) was loaded after some viscosity had built up. Samples prepared were numbered 8327-66-6, 7, 8, 9, 10.

[redacted] Samples were pulled on the Instron @ 0.5 in/minute.

	stress @ max load		stress @ max load
66-1	11.8 psi	66-6	30.2 psi
66-2	12.7 psi	66-7	89.9 psi
66-3	9.2 psi	66-8	63.6 psi
66-4	fell apart	66-9	81.4 [redacted] psi
66-5	8.9 psi	66-10	196.8 psi
ave	10.6 psi	ave	92.4 psi
std dev	1.9 psi	std. dev	62.7 psi

[redacted]

To Pag No.

Witnessed & Understood by m,	Date	Invent d by	Date
Brian Canelas	[redacted]		[redacted]
		Recorded by	

From Page No.

The Effect of Applying 40% EMB/60% NBD to a two part formulation. (5 + 10ml glass beads)

CONDITIONS

EMB	10.8g
NBD	25.2g
Europrene	6.35g
Cabosil	2.0g

PROCEDURE

EMB, NBD + Europrene were weighed into a glass jar and placed on a shaker for thirty minutes. 2g of Cabosil was added and shake for 5 minutes. This was Side A of the formulation. It was transfered to a 10:1 Cartridge (The A-Side).

330mg of grubb's Catalyst was dissolved in 5g of ~~pp~~ Dichloromethane (CH_2Cl_2) which was evaporated under flow of Nitrogen after 1.8g of Worthpar 100 processing oil was added. This resulted in a purple paste which was transfered to the B-Side of the Cartridge.

A 4" ~~Static Gun~~ and mixing tip and Static gun was used to dispense the formulation. (10ml glass beads)

OBSERVATION

fast gurning seem to be a problem and the paste was too foamy. Also the mixing tip seem to be too long. Decided to do 50:50 EMB/NBD + reduce the mixing tip.

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	

From Page No.

Repeat study of EMB/NBD to a two part formulation. (50:50) 5ml glass beads

CONDITIONS

EMB	18g
NBD	18g
Europrene	6.35g
Cabosil	2g

PROCEDURE Same as 8363-64

RESULT 5ml

SAMPLE ID	LOAD (psi)
836364A-1	79.30
" -2	114.50
" -3	74.87
" -4	98.24
" -5	113.90
	$\bar{x}$ 96
	σ 19

SAMPLE 10mL

SAMPLE ID	LOAD (psi)
836364B-1	100.9
" -2	96.2
" -3	58.9
" -4	113.9
" -5	101.2
	$\bar{x}$ 94
	σ 21

SAMPLE ID	LOAD (psi)
836365A-1	103.6
" -2	137.0
" -3	89.4
" -4	99.1
" -5	99.7
	$\bar{x}$ 106
	σ 18

OBSERVATIONS
All of the samples failed Adhesively

J. Jonathan Jones [redacted]

To Page No.

Witnessed & Understood by me, BQ

Date [redacted]

Invented by

Recorded by Jonathan Jones

Date [redacted]

From Page No.

The Effect of Grubbs Catalyst with Cyclopentene/Cyclohexene in a two part Adhesive formulation on Polypropylene Substrate. (10:1 Cartridge)

Formulations

A-Side	1A	2A
Norbornadiene	22.5g	22.5g
EMB	15.0g	15.0g
Blendex	7.5g	7.5g
Cabo-Sil	1.0g	1.0g
Zeospheres	4.0g	4.0g

B-Side	1B	2B
Grubbs Catalyst	0.385g	0.385g
Cyclopentene	4.615g	—
Cyclohexene	—	3.765g
Cab-O-Sil	—	0.10g
Blendex	—	0.75g

Procedure

A-Side formulation (1A&2A) were combined and mixed by hand in a plastic cup, but the blendex did not dissolve in the monomers (NBD/EMB). Europrene was substituted for Blendex, which was weighed in a bottle with the monomers. It was placed on a roller overnight, the formulation was successfully done the following day. A total of 2.4g of Cab-o-sil was used in the formulation.
The B-Sides were done separately.

Observations

The adhesive was found to be very fast cure, less than 15 seconds for 1B and less than 10 seconds for 2B. The 4" mixing tip had to be cut in half. We were able to get 6 samples for 1B and only 1 sample for 2B.

To Pag N

Witnessed & Understood by me,	Date	Invent d by	Date
Shawn McCollum	[redacted]	Recorded by Jonathan Taylor	[redacted]

Proj ct N .
B k N . 8374 TITLE RESULTS

Fr m Page No.

SAMPLE ID	LOAD (PSI)	FAILURE MODE
8374IB- 1	165.68	Coh/Adh
" - 2	298.84	
" - 3	230.16	
" - 4	268.36	
" - 5	243.84	
" - 6	187.00	

$\bar{x}$: 232
σ: 45

SAMPLE ID	LOAD (PSI)	FAILURE MODE
8374112B-1	123.38	Coh/Adh
" " -2		
" " -5		

J. Jan Jon

To Page No.

Witnessed & Understood by me,

Dat

Inv nted by

Rec rded by

Date

From Page No.

Repeat of 8374-11 using 1/10 the amount of Grubbs Catalyst on the B-Side. The A-Side stays the Same.

B-SIDE FORMULATION	1B	2B	A-SIDE
Grubbs Catalyst	~~35mg~~ ~~0.385g~~	35mg	Same as 8374-11
Cyclopentene	4.9g	—	
Cyclohexene	—	4.0g	
Cab-O-Sil	0.20g	~~0.20g~~ ~~0.185g~~	
Europrene		0.8g	

PROCEDURE Same as 8374-11

RESULT

SAMPLE ID	LOAD (psi)	FAILURE MODE
8374151B-1	—	
" -2	4.20	Adh/Coh
" -3	9.90	
" -4	7.90	
" -5	5.46	
	$\bar{x}$ 7	
	σ 2	

SAMPLE ID	LOAD (psi)	FAILURE MODE
8374152B-1	—	
" -2	10.82	Adh/Coh
" -3	6.20	
" -4	5.18	
" -5	4.98	
	$\bar{x}$ 7	
	σ 2	

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
[redacted]	[redacted]	Recorded by [redacted]	[redacted]

Project No._____
Book No. 8374 TITLE FORMULATION STUDY III

From Page No.___

The Effect of Grubbs Catalyst with Cyclopentene, Cyclohexene in a two part Adhesive formulation on polypropylene Substrate.

FORMULATION
~~Norbornadiene~~ JT

A - SIDE	AMOUNT	ACT AMOUNT (X10)
① Norbornadiene	22.5g	225g
② EMB	15.00g	150g
③ Europrene	7.5g	75g + 25g
④ Ca-bo-Sil	1.0g	10g
⑤ Zeopheres	4.0g	40g

B - SIDE

	1B	1B	2B	1B
Grubbs Catalyst	75mg	75mg	75mg	75mg
Cyclopentene	4.615g	—	—	2.308g
Cyclohexene	—	3.765g	3.765g	2.308g
Cabosil	—	0.10g	0.10g	0.10g
Europrene	—	0.75g	—	—

PROCEDURE Same as 8374-11

OBSERVATION
Grubbs JT catalyst and Cyclopentene cured in ~ 2 min resulting in a hard material. 50:50 Cyclopentene/Cyclohexene used instead.

T Pag No.___

Witn ss d & Underst od by me,	Dat	Inv nted by	Date
Shane Mell	[redacted]	Recorded by Jonathan Taylor	[redacted]

From Page No.

SAMPLE ID	LOAD (psi)	FAILURE MODE
8374222B-1	12.42	Adh / Coh
" -2	6.64	
" -3	10.82	
" -4	12.62	
" -5	—	
	$\bar{x}$ 11	
	σ 2	

SAMPLE ID	LOAD (psi)	FAILURE MODE
8374222B-1	19.60	Adh / Coh
" -2	5.20	
" -3	11.62	
" -4	9.10	
" -5	—	
	$\bar{x}$ 11	
	σ 5	

J. Taylor [redacted]

To Page No.

Witnessed & Understood by me, Shane Mell

Dat [redacted]

Invented by

Recorded by Josephine Taylor

Date [redacted]

From Page No.

The Effect of Grubbs Catalyst with Cyclohexene in a two part adhesive formulation on polypropylene substrate.

A-Side
Used 8374-22 (Masterbatch)

B-SIDE

Compound	Amount
Grubbs Catalyst	200mg
Cyclohexene	3.765g
Cabo-sil	0.20g
Europrene	0.75g

PROCEDURE
The Cyclohexene and Europrene were weighed into a small vial and placed on the paint shaker for 30 minutes. The mixture was added to the Grubb's Catalyst in a plastic cup, the Ca-bo-sil was added next and mixed well by hand before transferring to the Cartridge. Five samples were made and cured overnight in the hood.

RESULT

Sample ID	Time 16hr	Load (psi)	Failure Mode
83245[illegible]-1	"	101.86	Adh
" -2	"	56.64	"
" -3	"	141.98	"
" -4	"	180.00	"
" -5	"	312.60	Adh/Coh
		$\bar{x}$ 159	
		σ 87	

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
Shane M[illegible]	[redacted]	Recorded by Jonathan Taylor	[redacted]

Project N
Book No. 8374

TITLE (Cont'd)

From Page No.

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837451B-1	15 min	163.52	Adh
" -2	"	152.52	"
" -3	"	160.24	"
" -4	"	220.04	"
" -5	"	169.58	"
		$\bar{x}$ 173	
		σ 24	

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837451C-1	30 min	193.08	Adh
" -2	"	206.36	"
" -3	"	217.22	"
" -4	"	212.58	"
" -5	"	—	
		$\bar{x}$ 207	
		σ 9	

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837467D-1	1hr	230.84	Adh
" -2	"	232.62	"
" -3	"	257.92	"
" -4	"	278.76	"
" -5	"	—	
		$\bar{x}$ 250	
		σ 20	

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837451E-1	2hr	197.52	Adh
" -2	"	212.92	"
" -3	"	238.80	"
" -4	"	207.46	"
" -5	"	—	—
		$\bar{x}$ 214	
		σ 15	

To Page No.

Witnessed & Understood by me,

Date

Invented by

Date

Record d by

From Page No.

The Effect of Grubbs Catalyst with Cyclohexene in a two part Adhesive formulation on polypropylene substrate.

A-SIDE

80% Cyclal Methacrylate 20% Europrene

COMPOUND	AMOUNT
Cyclal Methacrylate	18.24g
Europrene	4.56g
Mono-2(Methacryloyloxy)ethyl Succinate	1.45g

B-SIDE

COMPOUND	AMOUNT
Grubbs Catalyst	200mg
NaEtOBEt3	1.29g (used 1.25g)
Ca-bo-sil	0.20g
Cyclohexene	3.76g
Europrene	0.50g

PROCEDURE

The components were added together separately in a small plastic cup, hand mixed for ~ 2-5 minutes and then transfered to the Cartridge. The Europrene and monomer were weighed into a small vial and placed on the paint Shakey for 30 minutes. A static gun and a 16" mixing tip was used to dispense the Adhesive on 20 polypropylene Samples.

OBSERVATION

The Samples were left to cure overnight as they were still not cured after 2 hrs. They were pulled on the speedy tester after allowing to sit for 1 week.

Result Next page

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Witnessed & Understood by me, [redacted] Date [redacted]

Invented by

Recorded by Jonathan Taylor Date [redacted]

Proj ct N .
B ok N .8374 TITLE RESULT (CONT'D)

From Page No.

RESULT

SAMPLE ID	LOAD (psi)	FAILURE MODE
837453- 1	85.32	Coh
" - 2	117.10	
" - 3	118.20	
" - 4	148.46	
" - 5	90.90	
" - 6	139.44	
" - 7	141.48	
" - 8	149.30	
" - 9	146.98	
" - 10	153.98	
" - 11	154.26	
" - 12	157.98	
" - 13	133.08	
" - 14	168.00	
" - 15	177.44	
" - 16	146.14	
" - 17	153.58	
" - 18	142.82	
" - 19	141.24	
	$\bar{X}$ 140	
	σ 23	

J. Taylor

To Page No.

Witn ssed & Understood by me, [redacted] Date [redacted]

Inv nted by

Date [redacted]

Record d by J. Taylor

From Pag No.

7-14-00

The effect of Grubbs Catalyst with Cyclohexene in a two part adhesive formulation on polypropylene substrate.

A- SIDE

Used 8374-22 (masterbatch)

B-SIDE

Compound	Amount
Grubbs Catalyst	200 mg
Cyclohexene	3.265 g
Ca-bo-sil	0.20 g
Europrene	0.75 g

PROCEDURE Same as in 8374-51

RESULT

Sample ID	Time	Load (psi)	Failure Mode	
837455A-1	5 min	277.62	Adh/Coh	samples were pulled between 5-10 minutes
-2		304.52	"	
-3		264.64	"	
-4		382.14	"	
-5		297.40	"	
		$\bar{x}$ 305		
		σ		

Sample ID	Time	Load (psi)	Failure Mode
837455B-1	10 min	147.90	Adh
-2		198.68	"
-3		492.56	Coh
-4		225.66	Adh
-5		270.40	"
		$\bar{x}$ 279	
		σ 120	

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Witnessed & Understood by me, [redacted] Date [redacted]

Invented by

Recorded by Jonathan Taylor Date [redacted]

Project No. _____

Book No. 8374 TITLE _____

From Page No. ___

Repeated 837450 at 5 minutes.

Sample ID	Time	Load (psi)	Failure Mode
837450C-1	5 min	112.3	Adh
" -2	"	315.2	Adh/Coh
" -3	"	113.4	Adh
" -4	"	151.2	"
" -5	"	362.1	Adh/Coh
		$\bar{x}$ 211	
		σ 106	

J. Taylor

To Page No. ____

Witnessed & Understood by me, _____ Date _____

Invented by _____ Date _____

Recorded by Jonathan Taylor

From Page No.___

The effect of Grubbs Catalyst with Cyclopentene.

A-SIDE
Used 8374-22 (masterbatch)

B-SIDE

Compound	Amount A	Amount B
Grubbs Catalyst	200mg	140 mg
Cyclopentene	4.8g	4.86g

Procedure — Same as in 8374-51

RESULT

Sample ID	Time	Load(psi)	Failure Mode	
837459A-1	1hr	81.50	Adh	Samples were pulled at 1hr-1:15
" -2	"	79.50	"	
" -3	"	98.34	"	
" -4	"	120.54	"	
" -5	"	63.66	"	
		$\bar{x}$ 89		
		σ 19		

Sample ID	Time	Load(psi)	Failure Mode
837459B-1	1hr	13.00	Adh
" -2	"	18.20	"
" -3	"	73.62	"
" -4	"	72.14	"
" -5	"	38.92	"
		$\bar{x}$ 43	
		σ 26	

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Witnessed & Understood by me,	Date	Invented by	Date
[redacted]	[redacted]	Recorded by Jonathan Taylor	[redacted]

[redacted]

60 Project No. ____ Book No. 8374 TITLE Metathesis Study (CONT'D)

From Page No. ___ [redacted]

The Effect of Grubbs Catalyst with Cyclohexene on B-Side and ENB on A-Side.

FORMULATIONS

A-SIDE Compound	Amount
ENB	37.5g
Europrene	7.5g
Zeeospheres	4.0g
Ca-bo-sil	1.0g

B-SIDE Compound	Amount
Grubbs Catalyst	200 mg
Cyclohexene	3.765 g
Ca-bo-sil	0.2g
Europrene	0.75g

A-Side formulation was prepared by adding the ENB and Europrene together in a 4oz glass jar and placed on the paint shaker; then the fillers were added.
The same was done on the B-Side.

RESULT

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837460-1	~~1 hr~~ 1.5 min	81.08	Adh
" -2	"	57.04	"
" -3	"	82.54	"
" -4	"	87.10	"
" -5	"	53.52	"
		$\bar{X}$ 72	
		σ 14	

J. Taylor
[redacted]

To Page No. ____

Witnessed & Understood by me, [signature] Date [redacted]

Invented by

Recorded by Jonathan Taylor Date [redacted]

From Page No.

The Effect of Super Grubbs Catalyst with Cyclohexene in a two part adhesive formulation on polypropylene substrate.

A-Side

Used Pomp Masterbatch 8374-22

Compound	Amount 8374-89A	B-Side's 8374-89B
Super Grubbs Catalyst	180 mg	50 mg
Cyclohexene	3.75 g	3.8 g
Ca-bo-Sil	0.20 g	0.20 g

* All = 11, 20, & 30 minutes study

PROCEDURE

Both B-Sides were mixed separately by hand in a small plastic beaker and then transfered to the Cartridge. Five Samples were made for each set. They were allowed to Cure over the week end.

Fine study was done with 8374-89A.

RESULT

Sample ID	All	Time (min) Load (psi)	Failure Mode	Comment
837489	All	1 " 16.20	Adh	
"	All	2 " 392.14	Coh	Completely Cured in < 5 min
"		-3 " 466.66	Adh/Coh	" "
"		-4 " 11.30	Adh	
"		-5 " 12.82	"	
		$\bar{x}$ 180		
		σ 205		

J. Jaynes

To Page No.

Witnessed & Understood by me, Shane Mk

Date

Invented by

Date

Record d by Jonathan Jaynes

Project No.
Book No. 8374 TITLE CONT'D

From Page No.

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE	COMMENT
837489A-1	20min	15.74	Adh	
" A-2	"	11.92	"	
" -3	"	446.24	Adh/Coh	Completely Cured in < 5min
" -4	"	454.40	"	" "
" -5	"	474.24	"	" "

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE	COMMENT
837489A-1	30min	385.96	Adh/Coh	Completely Cured in < 5 min
" -2	"	465.88	"	" "
" -3	"	465.10	"	" "
" -4	"	422.02	"	" "
" -5		$\bar{x}$ 432		
		σ 31		

SAMPLE ID	TIME (hr)	LOAD (psi)	FAILURE MODE	COMMENT
837489A-1	48+	43.38	Adh	
" -2		54.86	"	
" -3		229.62	Coh/Adh	Completely Cured in < 5 min
" -4		41.40	Adh	
" -5		42.70	"	
		$\bar{x}$ 82		
		σ 74		

SAMPLE ID	TIME (hr)	LOAD (psi)	FAILURE	COMMENT
837489B-1	48+	—	—	
" -2		18.26	Adh	
" -3		35.92	"	
" -4		28.02	"	
" -5		46.68	"	
		$\bar{x}$ 32		
		σ 10		

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
Shane M	[redacted]	Recorded by Jonathan Taylor	[redacted]